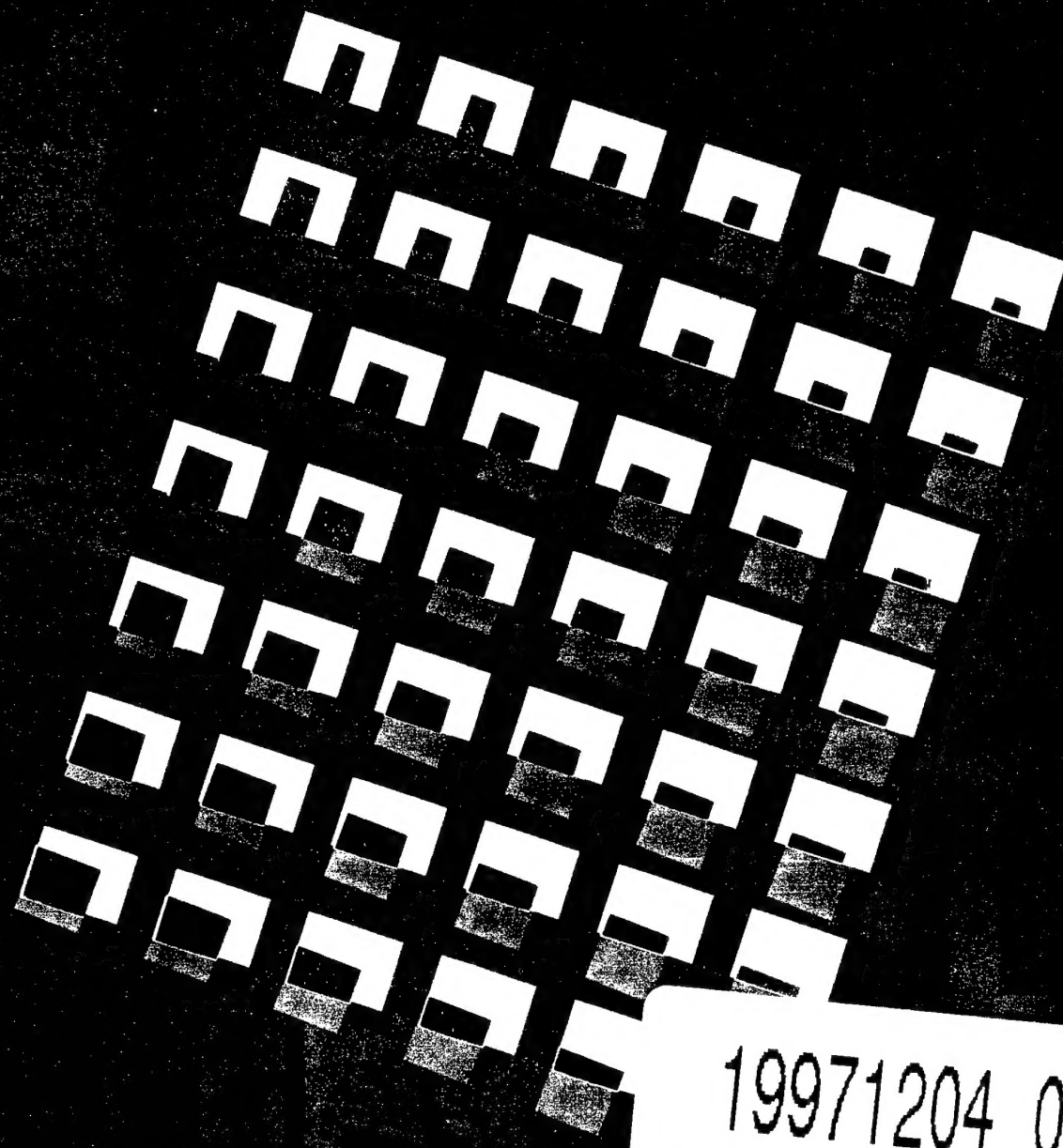


*TNO report*  
PML 1996-A87

**Strain rate results of the TNO-PML Cook-off test with HTPB/AP and PPG/AP/AN propellants**

TNO Prins Maurits Laboratory



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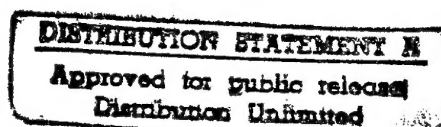
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## Managementuittreksel

Titel : Strain rate results of the TNO-PML Cook-off test with HTPB/AP and PPG/AP/AN propellants  
Auteur(s) : Ir. J.H.G. Scholtes  
Datum : september 1997  
Opdrachtnr. : A95KL408  
Rapportnr. : PML 1996-A87

Wereldwijd wordt veel aandacht besteed aan het begrijpen van het Cook-off fenomeen en de ontwikkeling van computermodellen die dit verschijnsel simuleren. De modellering van het complexe Cook-off mechanisme omvat onder andere de koppeling van thermische, chemische en mechanische computermodellen. Naast goede theoretische modellen en waarden voor de modelparameters, zijn gerichte testen voor de verificatie van de modellen nodig. De testen die echter op dit gebied uitgevoerd werden en worden zijn veelal gericht op de temperatuur en het tijdstip waarop de thermische weglooptreactie eindigt in een explosie. Naast de slecht gedefinieerde rand- en begincondities leveren deze testen geen data waarmee de bestaande modellen geverifieerd kunnen worden. TNO Prins Maurits Laboratorium (TNO-PML) is rond 1987 gestart met de ontwikkeling van een Cook-off test die het mogelijk moet maken het Cook-off probleem beter te kunnen bestuderen en de gewenste data te leveren.

Met de huidige TNO-PML Cook-off test kunnen, bij goed gedefinieerde randvoorwaarden en opwarmsnelheden, interne temperaturen van explosieve stoffen onder opsluiting gemeten worden. Omdat in het algemeen de responsie van een Cook-off reactie meer en meer van belang wordt is de aandacht in ons onderzoek verschoven naar 'het meten van de mate van heftigheid'. Onder opdrachtnummer A95KL408 is in 1995 gestart met de verdere ontwikkeling van de Cook-off test. In een eerste fase is getracht, met behulp van rekstroken, de responsie van de buis op een Cook-off of DDT-reactie te meten. Dit rapport beschrijft het vervolg van het onderzoek naar de meting van de heftigheid van een Cook-off reactie met rekstroken.

Er is een nieuwe serie geïstrumenteerde Cook-off testen met raketstuwstoffen uitgevoerd. De testen zijn uitgevoerd met 7 thermokoppels, 2 rekstroken en een drukopnemer. Globaal leveren de temperatuurmetingen goede, reproduceerbare resultaten die een goede indicatie zijn voor de interne radiale temperatuurverdeling in het testobject. Een niet geheel optimale temperatuurmeting van de wand in een enkele test zal in een volgende serie extra aandacht moeten hebben.

Een eerste poging om de interne druk tijdens het Cook-off proces te meten levert nog niet het verwachte resultaat op. De gemeten einddruk ligt ver onder de breekdruk van de buis die 240 MPa bedraagt. De druk werd gemeten met een drukopnemer die via een verlengstuk op een van de doppen gemonteerd was. De filterwerking van het verlengstuk kan een bijdrage leveren aan het onverwacht lage en vertraagde druksignaal. Een andere oorzaak moet gevonden worden in een blokkering van de stuwstof van de gasstroom voor het verlengstuk. Het probleem zou opgelost kunnen worden door enerzijds een poreus materiaal te gebruiken en anderzijds de gehele buis met doppen te isoleren. De isolatie zal resulteren in een hogere lokale temperatuur waardoor decompositie van de stuwstof zal optreden en de poreusiteit van het materiaal zal toenemen.

De nieuwe resultaten met de rekstroken zien er veelbelovend uit. De reproduceerbaarheid van de signaalgang in de laatste HTPB/AP-experimenten was goed. Indien de resultaten met de resultaten van de vorige serie experimenten bij elkaar genomen worden, kan een tendens waargenomen worden. Indien de gemeten responsie van een (Cook-off) reactie heftiger wordt neemt, zoals verwacht, de tijdafgeleide van de rek eveneens toe. Momenteel kunnen nog geen kwantitatieve uitspraken gedaan worden, maar we hopen in een volgende serie deze uitspraak te kunnen bevestigen en te kwantificeren. Hierbij moet een verbeterde drukmeting gekoppeld aan de rekmetingen een bijdrage kunnen leveren.

Het onderzoek om de TNO-PML Cook-off test te verbeteren zal in de volgende testseries voortgezet worden. Er zullen nieuwe experimenten met thermokoppels, rekstroken en drukopnemers uitgevoerd worden. Eveneens zal er getracht worden om een snelheidsmeting van de wand in het systeem te implementeren. Samen met de andere gemeten parameters zal dit ongetwijfeld een bijdrage leveren aan de validatie van de computermodellen en de oplossing van het Cook-off fenomeen waaraan wereldwijd onderzoek verricht wordt.

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## 1 Introduction

World wide, much effort is being put into Cook-off modelling of energetic materials in a confined geometry. Modelling a complex mechanism like Cook-off involves the coupling of thermal, chemical and mechanical computer codes. Besides good theoretical models, computer models and parameter values to be used in these models, experimental verification of these computer models is very important.

As described in our last report, 'Temperature and strain gauge measurements in the TNO-PML Cook-off test' [1], the main effort in our Cook-off research is concentrated on the measurement of the Cook-off response. This involves the measurement of parameters like strain and velocity of the cylinder wall and temperature and pressure inside the cylinder. Our first strain measurement results in DDT and Cook-off experiments were presented in this report. Because these results looked very promising, strain measurements in Cook-off experiments were continued. Also, a first attempt to measure the inside pressure build-up during a Cook-off with the absence of an ullage has been made. The experiments have been carried out with some newly developed PPG/AP/AN and HTPB/AP based rocket propellants. The results of these test series are presented in this report.

In Chapter 2 a short description of the experimental set-up is given. Chapter 3 describes the experiments, followed by the results. The results are split in three paragraphs, strain gauge, pressure and temperature measurements. The conclusions will be given in Chapter 4.

## 2 The experimental set-up

Because this test series is a continuation of the Cook-off strain gauge experiments, only a short description of the experimental set-up will be given. A more outlined description of several parts of the experiment set-up can be found in the reports [1] and [2]. In Figure 1, a construction drawing of the test cylinder is given. The strain gauge configuration for Cook-off experiments described in report [1] is used in the experiments. The strain gauges are positioned 2 cm from the centre of the tube at an angle of 180°.

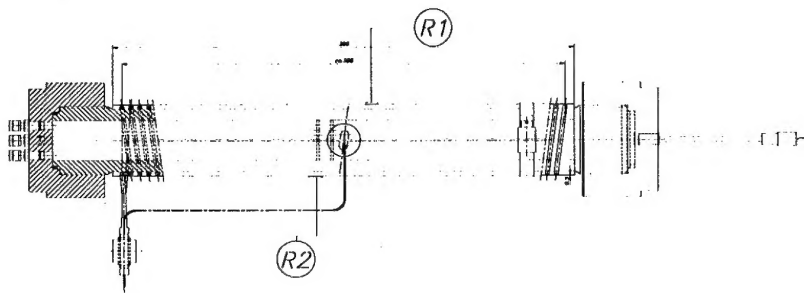


Figure 1: A construction drawing of the TNO-PML Cook-off test cylinder.

Temperatures were measured with 7 thermocouples for most experiments; 4 inside the cylinder at 0.0, 4.4, 8.8, 13.1 mm from the centre line of the tube, 2 at the wall and 1 at the outer edge of the cap. To get a good indication of the pressure build-up inside the tube, a Kistler 6211 pressure transducer with a range of 0-750 MPa was mounted to the test object. To protect the transducer against fragments and high temperatures, a coupling device is used (see Figure 1). The device will certainly have an influence on the signal, but this is not that important at the moment.

### 3 Experiments and results

#### 3.1 Introduction

In this test series, rocket propellants produced for a feasibility study of 'IM-igniters' have been used in the Cook-off experiments. The first type was a HTPB/AP propellant with a solid load of 85%. The second type was a PPG/AP/AN propellant with a solid load of 80.5% with 75.65 wt % Ammonium Perchlorate (AP) and 4.85 wt % Phase Stabilised Ammonium Nitrate (PSAN).

The preparation of the test series is about the same as in the first series of experiments. An accurate preparation of the strain gauges is very important. After the gauges have been glued onto the cylinder, the glue must be cured for a few hours in an oven at a temperature of at least 50 °C above the expected maximum temperature during the experiment. The rest of the preparation of the test is the same as described in the former report [1] and must be carried out accurately.

In two of the four experiments, a pressure transducer with the coupling devices was mounted to the cap and connected to the data acquisition system. The strain gauge and pressure measurement have been performed at a rate of 5 MHz. The thermocouples signals were sampled at a rate of 1 Hz.

#### 3.2 The strain gauge results

In Figure 2, the strain gauge results of the two PPG/AP/AN propellants are shown. The strain with a dimension of  $\mu\text{m}/\text{m}$  is given as a function of time. The time along the x-direction must not be seen as an absolute value but is the time relative and only important for the determination of the signal duration. The two signals on the right are the responses of two strain gauges in the first experiment; the left curve is the strain response of the second experiment, having a maximum strain of 15500  $\mu\text{m}/\text{m}$  and a maximum positive time derivative of 139  $\text{m}/\text{m}\cdot\text{s}$ . The other curves have a strain of respectively 12500 and 7000  $\mu\text{m}/\text{m}$  before breaking of the gauge and a slope of 350 and 89  $\text{m}/\text{m}\cdot\text{s}$ .



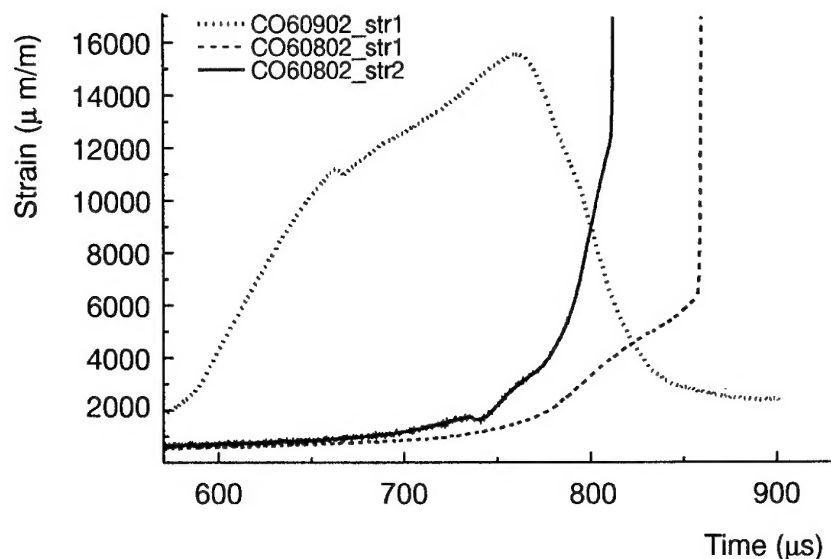


Figure 2: Strain gauge results of PPG/AP/AN Cook-off experiments.

An inspection of the fragments, after the first experiment, revealed that one of the strain gauges was exactly located on a split, while the opposite gauge was located in the middle of a fragment. Which signal is due to which gauge is not known, but it can be derived from the signal that the most right trace has a slope  $>10000 \text{ m/m}\cdot\text{s}$  after a strain of  $7000 \text{ }\mu\text{m/m}$ , which is only half of the maximum strain of this gauge type. It is not clear if the slope is due to the breaking process of the gauge or due to the bandwidth of the bridge amplifier during the fast bulging process. A fragmentation pattern of the tube used in this experiment is shown in Photo A.1 in Annex A.

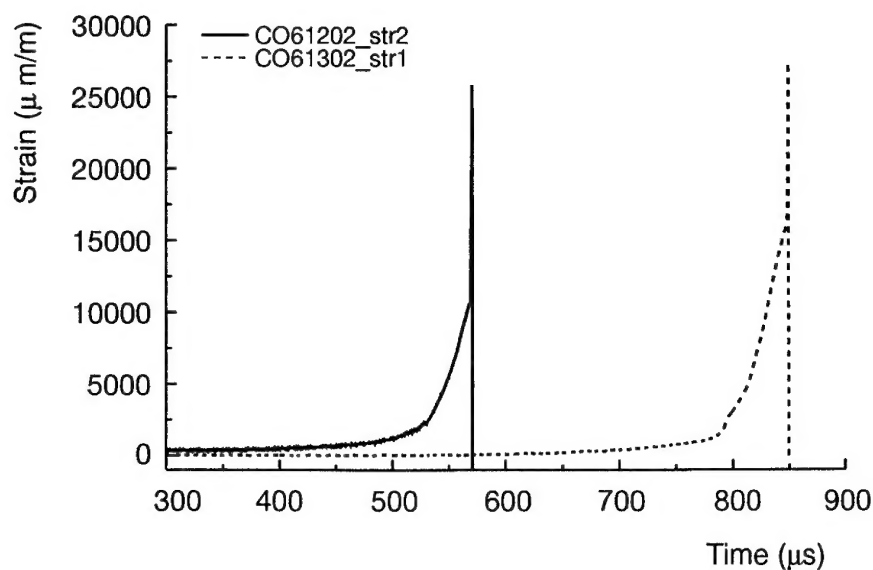


Figure 3: The Strain gauge results of HTPB/AP Cook-off experiments.

In Figure 3, the strain gauge results of the two HTPB/AP-based propellants are shown. Although these two signals are not exactly the same, their form is much more similar than the PPG experiments. In the left signal, the maximum strain is  $11000 \mu\text{m/m}$ , while the right signal has a maximum of  $16000 \mu\text{m/m}$ . The maximum time derivative of the left signal is  $350 \text{ m/m}\cdot\text{s}$  and the right has a derivative of  $400 \text{ m/m}\cdot\text{s}$ . The fragmentation of the tubes used in these two experiments is shown in Photos A.2 and A.3 in Annex A.

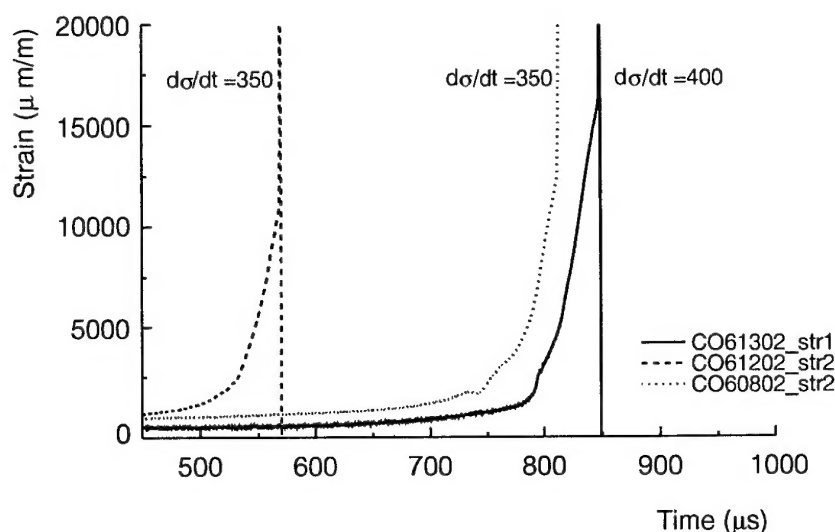


Figure 4: HTPB and PPG strain results with a derivative of 350 and 400  $\text{m/m}\cdot\text{s}$ .

For comparison reasons, the strain result of experiment co60802-str2 (see Figure 2) is added to the HTPB signals (Figure 4). This experiment also has a maximum derivative of  $350 \text{ m/m}\cdot\text{s}$  and a comparable fragmentation to the HTPB/AP experiment with a derivative of  $350 \text{ m/m}\cdot\text{s}$  (see Photos A.1 and A.2). The right most curve has a maximum derivative of  $400 \text{ m/m}\cdot\text{s}$  and indeed a response that is a little more severe than the two experiments with a derivative of  $350 \text{ m/m}\cdot\text{s}$  (see Photo A.3).

To compare more experimental results, the fragmentation of some experiments described in report [1] are shown in Annex A, Photos A.4 and A.5. Photo A.4 shows the fragmentation of a Cook-off experiment with AMPA. Comparing this fragmentation pattern with the fragmentation of the experiment with PPG and a derivative of 350 and  $400 \text{ m/m}\cdot\text{s}$ , we notice that the first response is more severe. The derivative of this experiment is about  $800\text{-}900 \text{ m/m}\cdot\text{s}$ . In Photo A.5 the fragmentation of the tube of a DDT experiment with Hexocire is shown. The DDT reaction is initiated from the left side and ends in a detonation at right side of the tube. The fragments at the left side are smaller than the fragments in the AMPA experiment so the response is a little more severe than the experiment, with AMPA. The corresponding strain rate of the tube in the DDT experiment is around  $1200 \text{ m/m}\cdot\text{s}$ . In this last photo, the fragments taken from the middle to the right

side of the cylinder are the product of a fast deflagration and even of a detonation. Exact values for the strain rate for this type of fragmentation are not yet measured but they are at least a few thousand m/m.s.

### 3.3 The pressure measurements

Only two Cook-off experiments were instrumented with pressure transducers. There was no ullage in the cylinder, only in the coupling device. In Figure 5, the result of the pressure measurement of the second PPG/AP/AN experiment is shown. The maximum pressure is 18 MPa. Compared to the strain gauge signal, the signal is 750  $\mu$ s later. The spikes on the signal are not understood. Presumably, the coupling device works like a filter for the pressure inside the tube. The steel cylinder can withstand 240 MPa, which is more than 10 times the measured pressure. So, the pressure measured is definitely not the real pressure inside the tube. Besides the filtering property of the coupling device, another cause can be responsible for the incorrect absolute value of the pressure. Although the PPG binder system starts to lose its integrity and mechanical strength beneath 100 °C, it is questionable whether the porosity of the propellant is sufficiently high that gas can flow into the coupling device to the pressure transducer. The propellant near the cap is expected to have a temperature comparable to the cap temperature measured (see Figure 8), which is about 70 °C. Therefore the propellant near the coupling device could act like a barrier for the gas flow.

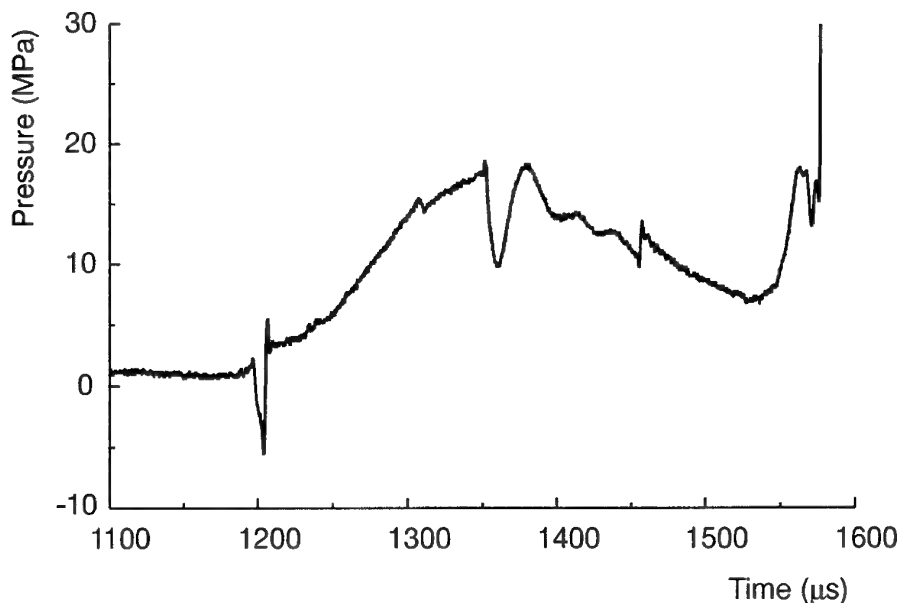


Figure 5: A pressure measurement of a PPG/AP/AN Cook-off experiment.

In Figure 6, the pressure measurement of the last experiment is shown. The maximum pressure in this HTPB/AP propellant experiment is 25 MPa. Compared to the

first pressure experiment, the pressure is higher, and derived from the fragmentation, its response is more severe. In this experiment, the peak pressure is measured 400  $\mu\text{s}$  later than the strain gauge peak. Also in this curve, spikes are superimposed on the pressure signal. The negative pressure signal is probably due to a breaking of the cable after the explosion. Also the pressure is much lower than expected, which is probably due to the blocking of the pressure by the propellant.



Figure 6: A pressure measurement of a HTPB/AP Cook-off experiment.

### 3.4 The temperature measurement

In this test series, the contact of the controller/wall thermocouple to the controller was not optimal. The influence of an imperfect connection is shown by a disturbance on the temperature signal at rising temperatures. After an initial heating period and at a constant heating rate  $k$ , the temperature distribution, in the radial direction of a cylinder, is time independent and has the form of a parabola ([1], Annex B). To have a good estimate of the 'correct' wall temperature, a straight line can be drawn parallel to the first inner temperature curve after the initial phase.

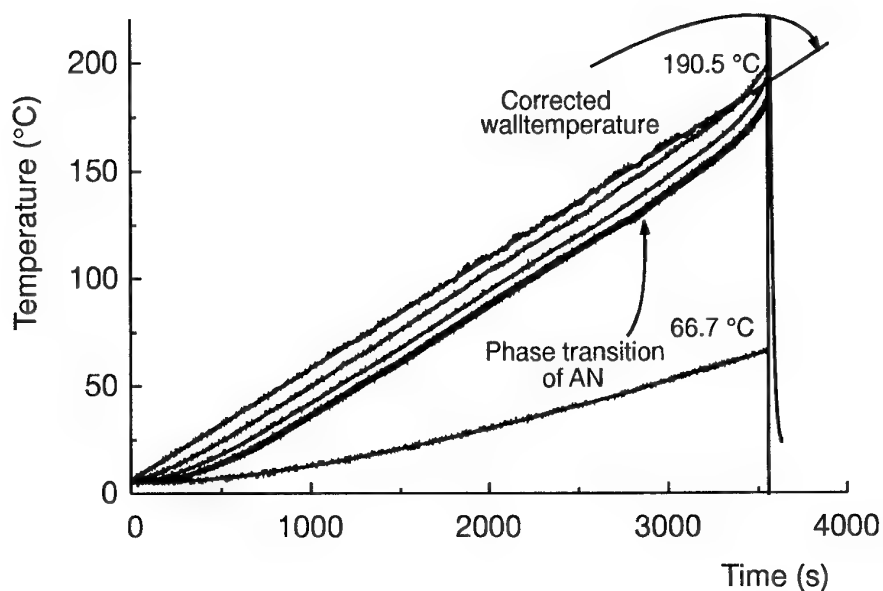


Figure 7: The temperature distribution of the first PPG/AP/AN Cook-off experiment.

In Figures 7 and 8, the temperature curves of the PPG/AP/AN experiment are shown. For the PPG experiment in Figure 7, a correction of the wall temperature was not necessary, but a straight line is drawn anyway. The temperature increase is constant after the initial phase. At 2800 seconds, the temperature of the thermocouple located in the centre (lowest curve in high temperature region) has a small fluctuation. This fluctuation is due to the crystalline phase transition of Ammonium Nitrate (AN) in this PPG propellant. The phase transition between phases II→I of AN takes place at a temperature of 125.2 °C [3]. Near the end phase of the experiment, the temperature increases faster than linear with time due to self heating of the propellant. The experiment ends in a mild explosion after 3550 second at a wall temperature of 190.5 °C. The fragmentation is shown in Photo A.1 in Annex A.

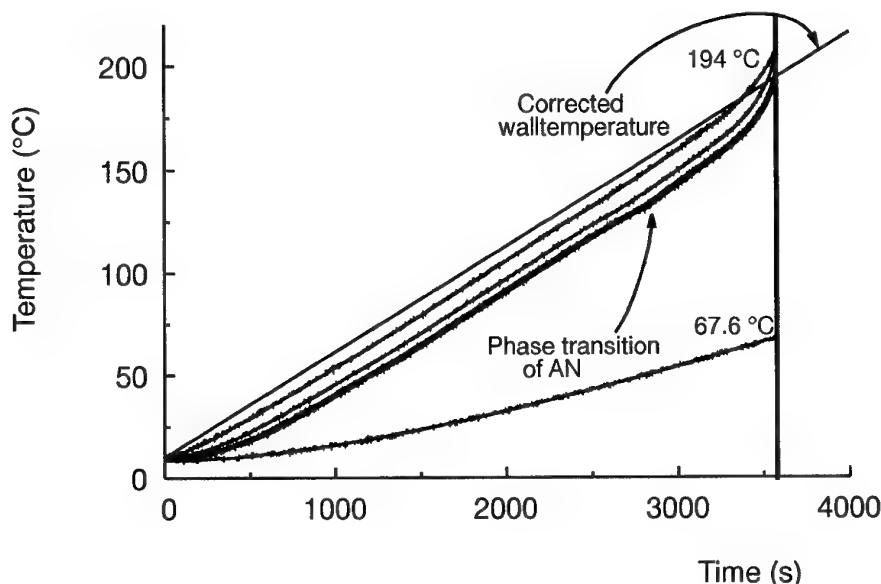


Figure 8: The temperature distribution of the second PPG/AP/AN Cook-off experiment.

In the second PPG/AP/AN experiment, in Figure 8, a corrected wall temperature is drawn. The temperature distribution is almost the same as in the first experiment. Also in this experiment, a temperature fluctuation at 125.2 °C is measured at 2800 seconds due to the crystalline phase transition. The experiment ends after 3570 seconds at a wall temperature of 194 °C, after a decomposition reaction of the propellant. The response is mild and the fragmentation is about the same as in the first PPG experiment.

The next two experiments were carried out with HTPB/AP propellants. The temperature distribution of the first experiment is shown in Figure 9. The temperature rise of this experiment has no real peculiarities. After an initial heating phase, the temperature rises linearly with time until the propellant starts to heat the test object. A runaway reaction is shown in the end phase of the experiment. The experiment ends after 3670 seconds at a wall temperature of 199.5 °C. The response of this experiment is also very mild and the fragmentation is shown in Photo A.2 in Annex A.

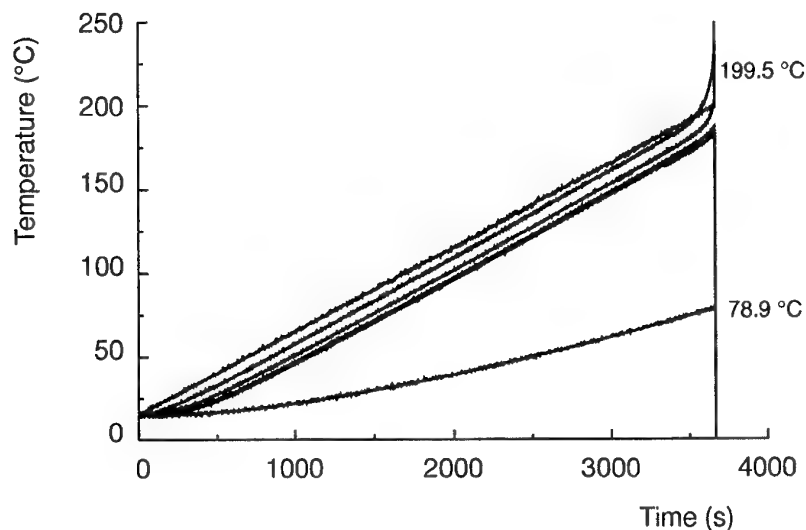


Figure 9: The temperature distribution of the first HTPB/AP Cook-off experiment.

The result of the temperature measurement of second HTPB/AP experiment (Figure 10) is not as good as the results of the first experiment. After some start up problems, which led to initial temperature differences, the temperature measured by the centre thermocouple followed a line different from the expected one, while the other internal thermocouple measured the expected linear temperature increase. The measured temperatures of this thermocouple are therefore questionable. The remainder of the experiment was as expected. After the self-heating of the propellant, the experiment ends at 3590 seconds and a wall temperature of 201.5 °C. Compared to the first HTPB/AP experiment, the response was a little more severe, deduced from the fragmentation (Photo A.3 Annex A), but the differences are very small.

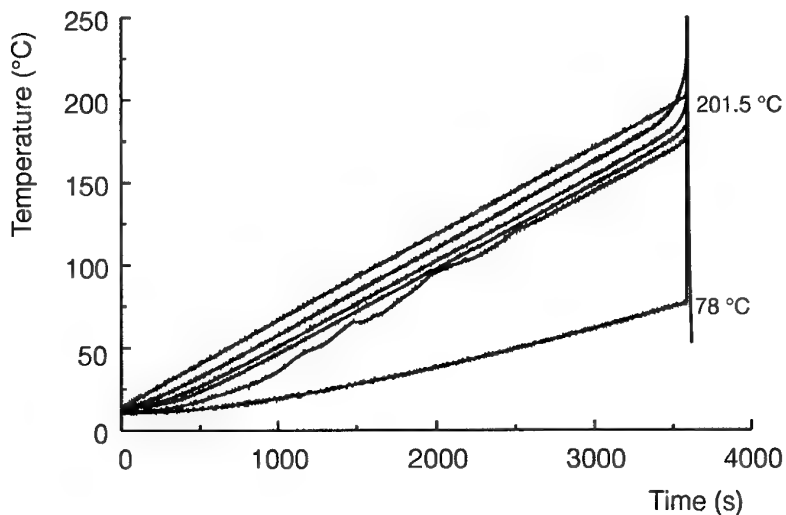


Figure 10: The temperature distribution of the second HTPB/AP Cook-off experiment.

## 4 Conclusions

A series of Cook-off experiments was carried out in the instrumented TNO-PML Cook-off test with newly developed propellants. The tests were instrumented with 7 thermocouples, 2 strain gauges and a pressure gauge.

The temperature measurements were reproducible and gave a good insight into the radial temperature distribution inside the cylinder. In one experiment in this series, the thermocouple connection to the cylinder wall was not optimal. This is a point of slight concern in future experiments.

Internal pressure measurements during the Cook-off runaway have been carried out. The pressure transducer was connected to the cylinder by a coupling device that has an influence on the pressure measurement. The pressure measured during the bulging process of the tube was far too low compared to the burst pressure of the tube (240 MPa). This could be the result of the coupling device acting as a filter on the pressure signal or of a non-porous propellant blocking gas flow to the pressure transducer (or both). This problem could be solved by temperature isolation of the cap. Due to higher local temperatures, the propellant in front of the pressure transducer will decompose as well, which results in an increased porosity. A porous energetic material instead of a cast energetic material should not show this problem.

The strain measurements in Cook-off experiments were continued. New promising results were found. The reproducibility of the signal form in the last two HTPB/AP experiments was good. Adding the strain gauge results in this series to the earlier results of the Cook-off and DDT experiments, a tendency in the results was assessed. If the response of a Cook-off reaction is more severe, a higher value of the strain rate is measured. At this moment, no quantitative values can be given, but we hope to confirm and quantify the present experiment results in future experiments. Finally, an improved internal pressure measurement coupled to the strain rate will produce a more quantitative value for the severity of an explosive reaction.

The research to improve the instrumented TNO-PML Cook-off test will continue in the following test series. New experiments with strain gauges, pressure transducers and thermocouple will be performed. Also an attempt to implement a velocity measurement of the wall will be made. Together with the other parameters measured, this will contribute to the validation of Cook-off computer models and to Cook-off problem solving all over the world.



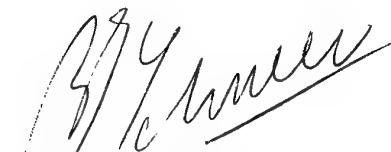
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J. Agr. Food Chem., 16 (1968) 373-377.

## 6 Authentication

A handwritten signature in black ink, appearing to read 'J.H.G. Scholtes', with a large, sweeping flourish extending from the bottom left.

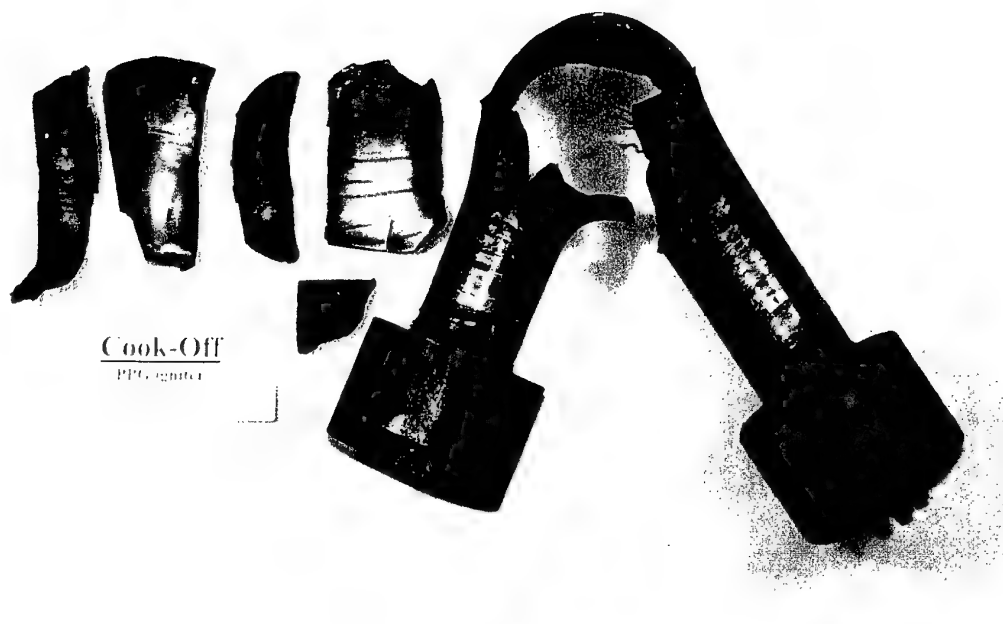
J.H.G. Scholtes  
Project leader/Author

A handwritten signature in black ink, appearing to read 'Dr. B.J. van der Meer', with a large, sweeping flourish extending from the bottom right.

Dr. B.J. van der Meer  
Research Co-ordinator

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## **Annex A      Photos of the fragmentation**



*Photo A.1: Fragmentation of a TNO-PML Cook-off test with a PPG/AP/AN propellant.*



*Photo A.2: Fragmentation of the first TNO-PML Cook-off test with an HTPB/AP propellant.*

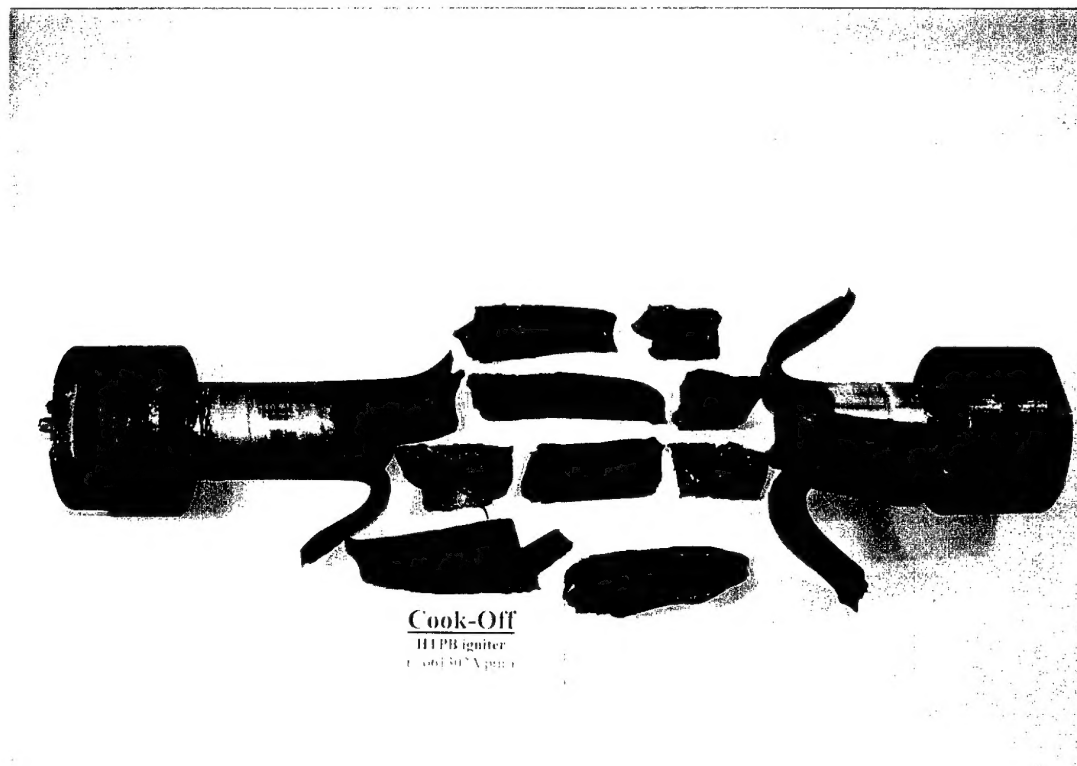
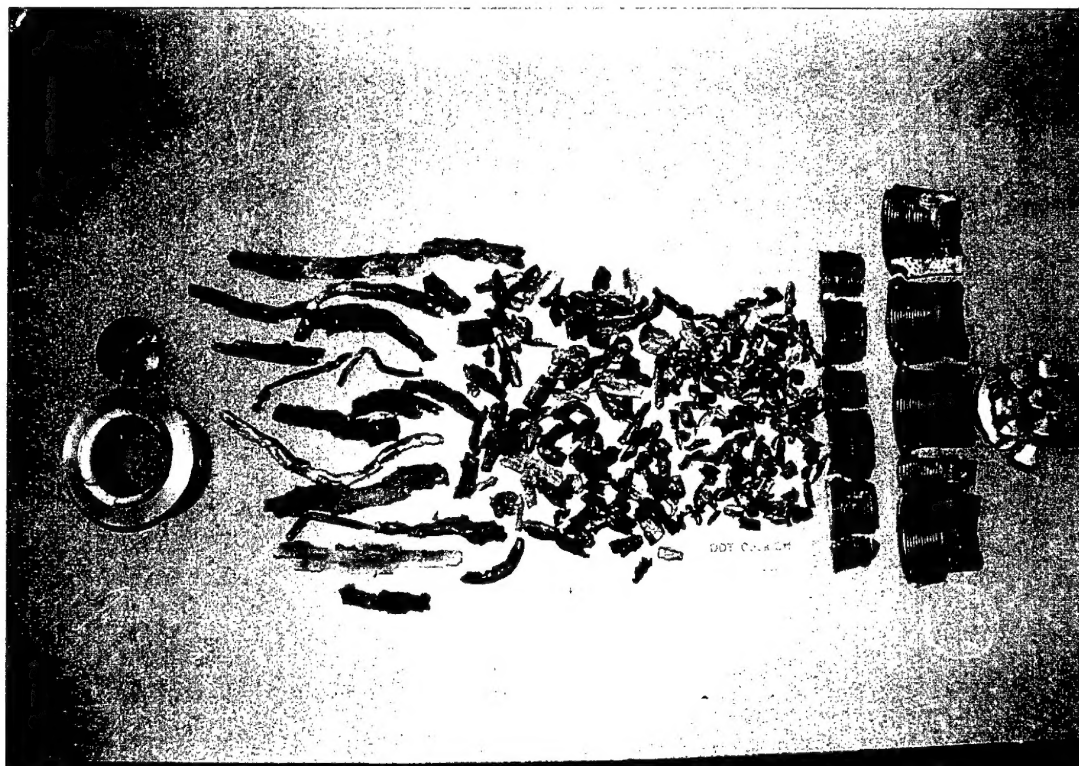


Photo A.3: Fragmentation of the second TNO-PML Cook-off test with an HTPB/AP propellant.



Photo A.4: Fragmentation of a TNO-PML Cook-off test with AMPA.



*Photo A.5: Fragmentation of a DDT test with the TNO-PML Cook-off test with Hexocire. The reaction was initiated by a pyrotechnical mixture from the left side.*

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<b>15. ABSTRACT (MAXIMUM 200 WORDS (1044 BYTE))</b> <p>After the first series of Cook-off experiments with strain gauges, the development of the TNO Prins Maurits Laboratory (TNO-PML) Cook-off test continues. A series of new Cook-off experiments has been carried out with newly developed propellants. The results of the temperature measurements are good and give a good idea of the radial temperature distribution inside the cylinder. The first pressure measurement during the Cook-off runaway did not give the result expected. Probably, a coupling device and the blocking of the propellant at its opening led to a signal more than 10 times lower than the burst pressure of the cylinder. An isolation of the caps or the use of porous materials could solve this problem. The strain measurements gave some new promising results. The reproducibility of the signal-form in the last two experiments was good. It can be stated that a higher value of the strain rate is measured if the response of a (Cook-off) reaction is more severe. Quantitative values cannot be given yet, but we hope to confirm and quantify the statement in future. Future experiments with improved pressure measurement coupled to the strain rate will produce a more quantitative value for the severity of an explosive reaction. Together with an attempt to measure the wall velocity of the wall, this will definitely contribute to the validation of Cook-off computer models and problem solving of the Cook-off phenomenon all over the world.</p>				
<table style="width: 100%; border: none;"><tr><td style="width: 50%; vertical-align: top;"><b>16. DESCRIPTORS</b> Strain rate Cook off Rocket propellants Temperature measurements</td><td style="width: 50%; vertical-align: top;"><b>DESCRIPTORS</b> Pressure measurements Strain measurement Experimentation Computer programs</td></tr></table>			<b>16. DESCRIPTORS</b> Strain rate Cook off Rocket propellants Temperature measurements	<b>DESCRIPTORS</b> Pressure measurements Strain measurement Experimentation Computer programs
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